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In re application of: Kenneth M. Fyles	s and Helen L. McPhail
Serial No.: 08 / 190,883 Filed: February 3, 1994 For: GLASS COMPOSITIONS	Group No.: 1108 Examiner:
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Attached please find the certified copies are claimed for this case:	s of the foreign applications from which priorities
Country: United Kingdom	•
Application Number: 9302186.3	
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O Title of invention

 Please give the title of the invention

NEUTRAL COLOURED GLASSES

@ Applicant's details

- ☐ First or only applicant
- 2a If you are applying as a corporate body please give:

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Neutral Coloured Glasses

The present invention relates to infra red (IR) and ultra violet (UV) absorbing soda lime silica glasses for use in glazing. more particularly it relates to windows of a neutral tint made of such glasses, particularly for vehicles including motor cars.

Special glasses have been developed for use in vehicles with a low level of direct solar heat transmission (DSHT) and ultra violet transmission (UVT) so as to reduce the problems caused by excessive heating within the vehicle on sunny days, and to protect the interior furnishings of the car from the degradation caused by ultra violet radiation. Glasses with good infra red absorption are usually produced by reducing iron present in the glass to the ferrous state or by adding copper. Such materials give glasses a blue colour. The materials added to achieve good ultra violet radiation absorption are Fe^{3+} , Ce, Ti or V. The quantities added to cause the desired level of absorption are such as to tend to colour the glass yellow. The combination in the same glass of both good UV and good IR absorption gives glasses whose colour is either green or blue. This means that when the colour of the glasses is defined by the CIELAB system, the commercial glasses in 4 mm thickness which have greater than 60% transmission are found to be either very green -a*>8 or very blue -b*>7.

Proposals have been made to produce vehicle glazing with good

protection against IR and UV radiation in grey or bronze, but the proposed glasses tend to have a greenish yellow tinge. Thus in, for example, French 2.672.587, the dominant wavelength (λ D) of the glasses exemplified varies from 571 to 580 nm, and the colour purity is in the range 4.4 to 15.9% indicating a greenish yellow tinge. A commercial neutral grey glass, ANTISUN (ANTISUN is a trade mark of the Pilkington Group) Grey, available in commerce from Pilkington Glass Limited of St. Helens, England, has a dominant wavelength of 454 and a colour purity of 2.1% in a thickness of 4 mms.

We have identified a requirement for glasses with a neutral tint such that, in the CIELAB system, they have a colour co-ordinate range a from -6 to +5, b from -5 to +5, at visible transmissions above 60% and a* -12 to +5, b* -5 to +10 below 60% visible transmissions (hereinafter referred to as a neutral tint as herein defined)..

We have found that glasses with a neutral tint free from any dominance in the range 570 nm to 580 nm, and with visible light transmissions greater than 32% (at a thickness of 4 mm), can be formed with a direct solar heat transmission which is at least 7 percentage points (preferably 10 percentage points) less than the % transmission of visible light by using a specified total iron content, a specified minimum FeO content and neutralising the resulting green or blue tint with one or more of Se. Co₃O₄, Nd₂O₃, NiO and MnO. The glasses have a dominant wavelength which is

preferably less than 570. The colour purity for a glass thickness of 4 mm, is preferably not greater than 4 and especially less than 2, (especially when the dominant wavelength is above 570 nm).

Our invention is based on the surprising discovery that the incorporation of relatively large amounts of other specified colouring agents compensates for the green colour arising from the presence of infra red and ultra violet radiation absorbing components. but that such amounts can be incorporated at the levels required without causing an unacceptable reduction in light transmission.

French Patent Specification 2,672,587 at page 5 lines 32 to 35 points out that both CoO and NiO reduce visible light transmission without contributing to either the absorbtion of ultra violet or infra red radiation, and that it is preferable not to add these components. An upper limit of 0.005% is suggested for CoO but the highest CoO content exemplified is .0010%, supporting the teaching in the specification that these materials should not be used and, if they are, should be present only in limited quantities.

In French Patent 2.672.587, the quantity of Se used in the examples tends to be of the same order or greater than the amount of tinting agent except for one example, example 9, where the ${\rm Fe_20_3}$ content is low at 0.178 and the colour purity has a high numerical value 9.6, indicating a substantial departure from a neutral appearance for a 4 mm glass of transmission greater than

60%. We have found it necessary to limit the amount of Se so that it is much less than the other tinting agents.

The field of tinted glasses is one in which relatively small changes can produce major changes in tint. As one patentee has said, it is like a hunt for a needle in a haystack. Wide ranges disclosed in prior patents can encompass many possibilities, and it is only the teaching of the specific examples that can be relied on as identifying how particular tints associated with particular ranges of absorption of infra red and ultra violet radiation can be obtained. It is clear that in French Patent 2.672.587, the correct balance of components to achieve a neutral tint free of unacceptable dominance at wavelengths above 570 nm has not been taught. In attempting to get relatively low DSHT, French patent uses high levels of FeO and clearly it has not been recognised, as has been discovered in the present invention, that this can be balanced by addition of tinting agents without an unacceptable loss in visible light transmission.

According to the invention, there is provided, an IR and UV absorbing soda lime silica glass of a neutral tint as herein defined having a ferrous iron content:

% by weight FeO \geq 0.007 + (Optical Density - 0.036) 2.3

and a total iron content expressed as Fe_2O_3 in the range 0.25-1.75% by weight, the glass having been tinted to a neutral colour by one or more of Se, Co_3O_4 , Nd_2O_3 , NiO and MnO, the glass having, in 4 mm thickness, a visible light transmission of at

least 32%, a UV transmission preferably less than 25%, and a direct solar heat transmission at least 7 percentage points (preferably 10 percentage points) below the visible light transmission, and having a dominant wavelength preferably less than 570 nm; provided that, when the dominant wavelength is above 570 nm and the visible light transmission is greater than 60%, the colour purity is not greater than 4, and preferably less than 2, and that when Se is present, at least one other colourant selected from Co₃0₄, Nd₂0₃. NiO and MnO is present in an amount by weight at least 1.5 times the amount of Se present, preferably 2 times as much.

The quantity of Se is preferably kept as low as is compatible with getting a satisfactory neutral colour. We find it possible to operate satisfactorily at less than 5 ppm Se at visible transmissions greater than 60%.

For the purpose of the present specification and claims, references to visible light transmission are to light transmission (LT) measured using CIE Illuminant A; references to UVT are reference to transmissions measured in accordance with the international ISO 9050 standard over the wavelength range 280 to 380 nm; references to direct solar heat transmission (DSHT) are references to solar heat transmission integrated over the wavelength range 350 to 2100 nm according to the relative solar spectral distribution Parry Moon for air mass 2; total solar heat transmission (TSHT) equals DSHT plus the solar heat that is absorbed and reradiated in the forward direction.

Figure 1 is a plot of transmission against FeO content for a series of grey glasses showing the improvement in properties that can be achieved, in accordance with the invention, by increasing the FeO content of a known glass (glass 1) while incorporating colourants, as taught in the present invention, to maintain a neutral tint. Glass 1 is commercially available grey glass with a DSHT similar to its light transmission and a UV transmission about 40%. In glasses 2, 3 and 4 the amount of ferrous ion has been increased which, without compensating changes, would normally turn the glass blue and significantly reduce light transmission. The UV transmission has also been reduced which would normally introduce a greenness to the blue glass. The light transmission and neutrality of the glass have in fact been maintained by a balance of cobalt, neodymium, and selenium additions while controlling the ferrous/ferric ratio.

Selenium introduces a pink component to the colour of the glass thus complementing any blue in the glass to produce a more neutral colour. However, selenium also has colourless forms in oxidised glasses whilst reduced glasses are assumed to contain brown or colourless polyselenides. The oxidation/reduction of the glass must therefore be carefully controlled to retain selenium in the coloured pink form.

Selenium retention is at a maximum at about 20% ferrous/ferric ratio and is significantly reduced below 10% ferrous/ferric or above 40% ferrous/ferric. In glasses using selenium as a neutralising agent the ferrous/ferric ratio should

be between 10 and 40% for the best efficiency of retaining selenium in the coloured form.

Although cobalt in itself colours glass blue, it is useful as a colour neutralising agent because its absorptions are in the red end of the visible spectrum and can therefore be useful in counteracting the effect of reduced iron absorption in the infra red at 1050 nm.

Neodymium oxide is also useful in a similar way but is a better neutraliser than cobalt since it is dichroic and produces a blue and pink colouration depending on the kind of light in which it is viewed. However, neodymium oxide is expensive and cobalt in combination with selenium is preferred as a neutralising agent.

For glasses with a DSHT at least 7% points lower than the visible light transmission and a UV transmission of less than 25% in 4 mm path length, the following combination of additives is required.

Total iron expressed as Fe_2^{0} in the range 0.25-1.75%, usually 0.25-1.25%.

The minimum FeO content to provide the necessary DSHT varies with optical density according to the equation

% FeO
$$\geq$$
 0.007 + (optical density - 0.036)

where optical density = \log_{10} T/ $_{100}$ where T is the visible light transmission in percent for 4 mm glass.

This roughly equates to:-

Light	Transmission	Minimum	Fe0	Content
	80%	0.0	33%	
	70%	0.0	58%	
	60%	0.0	87%	
	50%	0.1	.22%	
	40%	0.1	53%	
	30%	0.2	18%	

Ultraviolet absorption will be provided by iron oxide in the ${\rm Fe^{3^+}}$ state, optionally supplemented by ${\rm V_2O_5}$ and/or, ${\rm CeO_2}$ and/or ${\rm TiO_2}$. For most tints, a UV transmission of less than 25% is met simply by ferric iron. Where the ferric iron is less than 0.3% expressed as ${\rm Fe_2O_3}$. UV transmission may be reduced below 25% by additions of:-

 $0.1 - 1.0 \text{ CeO}_2$ (if vanadium is absent)

0.05 - 0.2 v_2^0 (if cerium is absent)

Many of the glasses contain at least 0.3% ferric iron expressed as ${\rm Fe_20_3}$ and in addition have quantities of ${\rm Ce0_2}$ and ${\rm V_20_5}$ to provide enhanced UV protection.

Preferably the DSHT shall be at least 10% points lower than the visible light transmission; the minimum FeO content of the glass to meet this preferred condition is given by

%Fe0
$$\geq$$
 0.012 +(optical density - 0.036)
1.84

This roughly equates to:

Light Transmission	Minimum FeO Content
80%	0.045%
70%	0.076%
60%	0.113%
50%	0.156%
40%	0.208%
30%	0.276%

For the most neutral tints the ferrous/ferric ratio shall not be less than 10%.

A required tint may be achieved by adding one or more of Se, ${\rm Co_3O_4}$. ${\rm Nd_2O_3}$. NiO or MnO. The quantities of colourant and the colourant chosen to decolourise depends on the depth of tint required and the following guide lines are given.

Se up to 50 ppm chemical Se retained Co_3O_4 up to 200 ppm Nd_2^2O_3 up to 2.5%

Nickel oxide may be added as a colour neutralising agent but it is preferably limited to a maximum of 200 ppm to reduce the possibility of nickel sulphide inclusions which can shatter the glass.

Manganese oxide may also be added to the glass to provide pinkness but it is preferably limited to a maximum of 1% by weight

because of the danger of colour changes caused by solarisation.

Similarly, cerium and vanadium oxide ultra violet absorbers in the same glass are preferably avoided because of the possibility of colour changes by solarisation.

Other components which may be present in glasses in accordance with the invention include copper oxide, CuO typically in an amount up to 0.1% by weight, which in certain conditions may reduce solar heat transmission, and chromium oxide, ${\rm Cr_2O_3}$, typically in an amount up to 50 ppm, for colour modification.

The glasses in accordance with the invention are useful for both architectural and automotive purposes, and the invention also provides windows composed of glass in accordance with the invention. The automotive windows may be not only windscreens, but also the remaining windows of a car. These can eg include rear side windows with visible light transmissions as low as 30% and a rear screen as low as 50%.

The following examples except Example 1 (which is for comparison) illustrate but do not limit the invention. In the Examples, all parts and percentages are by weight and:.

- (a) Fe $_2$ 0 $_3$. Fe0, Nd $_2$ 0 $_3$. Ce0 $_2$. Ti0 $_2$. V $_2$ 0 $_5$. Sn0 and Mn0 $_2$ are expressed in percent; Se, Co $_3$ 0 $_4$ and NiO are expressed in parts per million.
- (b) total iron is expressed as if all iron present were present as ferric oxide.
- (c) percentage of total iron as Fe^{2+} is calculated from the spectral curve of the glass using the equation:-

$$^{\text{xFe}^{2+}} = 115.2 \text{ (OD}_{1000} - 0.036)}$$

where OD_{1000} = optical density of the glass at 1000 nm wavelength

t = glass thickness in millimetres

 Fe_2O_3 = percentage total iron, expressed as Fe_2O_3 , in the glass

(d) the FeO content is calculated from the equation

%Fe0 =
$$\frac{\text{%Fe}^{2+}}{100}$$
 × Fe₂0₃ × $\frac{143.7}{159.7}$

 Fe_2O_3 = percentage total iron, expressed as Fe_2O_3 , in the glass

(143.7 being the molecular weight of 2 \times FeO and

159.7 being the molecular weight of Fe_20_3).

Examples

CHEMICAL ADDITIVES

		Total iron as Fe ₂ 0 ₃	Fe0	% of total iron as Fe2		co ₃ 0 ₄	Nd ₂ 0 ₃	CeO ₂	TiO ₂	^V 2 ⁰ 5	Other
		*	*								
1	Solargrey	0.25	0.043	18%	11	41	-	-	-	-	-
2		0.35	0.082	26%	3	32	0.15	0.4	-	-	-
3		0.38	0.078	23%	5	32	-	0.34	-	-	-
4		0.38	0.099	29%	2	32	-	0.4	-	-	-
5		0.25	0.038	17%	9	_	-	-	-	0.1	-
6		0.25	0.065	29%	3	-	-	-	-	0.1	-
7		0.34	0.098	32%	۲2	32	-	0.4	-	-	-
8		0.45	0.030	7.5%	24	18	-	0.4	-	-	-
9		0.45	0.093	23%	5	38	-	0.4	-	-	-
10		0.45	0.142	35%	⟨2	38	-	0.4	-	-	-
11		0.45	0.154	38%	۲2	38	-	0.4	-	-	-
12		0.45	0.117	29%	۲2	-	0.2	0.4	-	_	-
13		0.45	0.126	31%	۲2	-	0.5	0.4	-	-	-

ģ,

	Total iron as Fe ₂ 0 ₃ Fe0	% of total iron as Fe2 ⁺ Se	^{Co} 3 ⁰ 4	Nd 2 ⁰ 3	CeO ₂	TiO ₂	v ₂ 0 ₅	Other
14 15 16 17	% % 0.45 0.122 0.45 0.142 0.45 0.142	30% <2 35% <2 33% <2 35% <2	38	0.3	0.4 0.5 0.4 0.4	- 0.25 0.2	- - -	- - - NiO
18 19 20 21	0.45 0.146 0.45 0.130 0.4 0.104 0.45 0.150	36% 5 32% 16 29% 8 37% 10	25 30 38	0.3	0.4 0.4 0.4 0.4	- - -	- - -	100ppm - - - - Sn0
22 23 24 25 26 27 28 29 30 31 32 33 34 35 36	0.675 0.249	41% 3	18 20 - 38 - 76 80 57 104 57 80 120 120	0.7	0.2 1.0 - 1.0 0.1 - - -	0.5	0.1	1.0
37 38 39 40	0.7 0.290 0.8 0.209 1.0 0.225	29% 24	20 Zero 81 150 20	- 2.0 - -	- - - 0.4	- - - -	- - -	- - - - MnO ₃
41 42		30% <2 11% 15 1	20 (120	0.1	- -	-	- -	1.0 ² NiO
43 44 45	1.23 0.277 2		20 104 .04	- -	-	- - -		275ppm - -

TRANSMISSION COLOUR

	<u>LT</u>	DSHT	UVT	<u>a*</u>		ominantuelengt	t Colour th <u>Purity</u>
1 Solargrey 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44 45	7698820304442134477574091713815009937410633594269	701 96 59 75 58 6 4 5 5 5 4 5 4 7 6 4 7 7 7 4 8 5 0 1 2 5 6 6 9 5 7 6 9 8 7 6 0 9 5 8 4 7 7 7 4 8 5 0 1 2 5 6 6 9 5 7 6 9 9 5 8 4 2 2 2 2 2 6 0 9 5 8 4 7 7 7 4 8 5 0 1 2 5 6 6 9 5 7 6 9 5 8 4 7 7 7 8 5 0 1 2 5 6 6 9 5 7 6 0 9 5 8 4 7 7 7 8 5 0 1 2 5 6 6 9 5 7 6 0 9 5 8 4 7 7 7 8 5 0 1 2 5 6 6 9 5 7 6 0 9 5 8 4 7 7 7 8 5 0 1 2 5 6 6 9 5 7 6 9 5 8 7 6 0 9 5 8 4 7 7 7 8 7 8 7 8 7 8 7 8 7 8 7 8 7 8	40 23 21 22 17 23 25 23 20 22 23 20 22 18 15 20 19 18 22 11 21 8 12 12 13 14 15 16 15 19 11 16 15 17 17 18 18 18 18 18 18 18 19 19 19 19 19 19 19 19 19 19 19 19 19	-0.1.8.4.2.5.5.4.4.7.9.0.0.4.3.6.5.3.0.3.9.7.8.0.4.7.8.8.0.8.5.7.0.3.4.6.5.6.9.0.8.0.5.7.4.2.1.0.1.4.3.2.4.5.0.3.4.6.5.6.9.0.8.0.5.7.4.2.1.3.4.4.2.1.3.2.4.5.0.3.4.6.5.6.9.0.8.0.5.7.4.2.4.2.4.2.4.2.4.2.4.2.4.2.4.2.4.2.4	1.406952659826660370813075911221599333344045431 -12.695265982666603708130759112215993333440454341 +11.32.16.66603708130759111215993333440454341 +12.695266660370813075911121599333344045454341 +13.38.5942110045440454404544045440454404544045464045464045464546	4 5 7 8 9 5 5 4 4 5 4 5 5 5 5 5 5 5 5 5 5 5 5 5	2.7% 2.7% 2.7% 2.7% 2.7% 2.7% 2.7% 2.7% 2.9%

Several of the examples given are suitable as high performance architectural glasses for buildings because of their superb infra-red and UV absorbance combined with neutrality of colour. The properties of architectural glasses are often quoted in 6 mm path length and the Total Solar Heat Transmission (TSHT) is quoted in addition to the DSHT. Some examples particularly suitable for architectural use are as follows:-

Transmission	in	6	mm	path	length
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Example	Light Transmission	DSHT	TSHT	<u>UVT</u>
3	58%	48%	60%	15%
7	65%	48%	60%	20%
11	55%	36%	51%	17%
13	63%	44%	57%	16%
14	65%	44%	57%	16%
21	63%	44%	57%	16%

Claims

1. An IR and UV absorbing soda lime silica glass of a neutral tint as herein defined having a ferrous iron content % by weight FeO \geq 0.007 + (Optical density - 0.036) 2.3

and a total iron content expressed as Fe_2^{0} in the range 0.25 - 1.75% by weight,

the glass having been tinted to a neutral colour by one or more of Se, ${\rm Co_3O_4}$, ${\rm Nd_2O_3}$, NiO and MnO,

the glass having, in a 4 mm thickness, a visible light transmission of at least 32%, a UV transmission preferably less than 25%, and a direct solar heat transmission at least 7 percentage points below the visible light transmission, and having a dominant wavelength preferably less than 570 nm, provided that, when the dominant wavelength is above 570 nm and the visible light transmission is greater than 60%, the colour purity is not greater than 4,

and when Se is present, at least one other colourant selected from ${\rm Co_3O_4}$, ${\rm Nd_2O_3}$. NiO and MnO is present in an amount by weight at least 1.5 times the amount of Se present.

2. An IR and UV absorbing glass according to claim 1 having, in 4 mm thickness, a direct solar heat transmission at least 10 percentage points below its visible light transmission. A.

- 3. An IR and UV absorbing glass according to claim 1 or claim 2 having, in 4 mm thickness, a dominant wavelength above 570 nm, a visible light transmission greater than 60% and a colour purity less than 2.
- 4. An IR and UV absorbing glass according to any of the preceding claims containing Se and at least one other colourant selected from ${\rm Co_3O_4}$, ${\rm Nd_2O_3}$, NiO and MnO in an amount by weight at least twice the amount of Se present.
- 5. An IR and UV absorbing glass according to claim 4 having a visible light transmission greater than 60% and an Se content of less than 5 parts per million by weight.
- 6. An IR and UV absorbing glass according to the preceding claims containing 0.1 to 1.0% by weight ${\rm CeO}_2$.
- 7. An IR and UV absorbing glass according to any of claims 1 to 5 containing 0.05 to 0.2% by weight ${\rm V_2O_5}$.
- An IR and UV absorbing glass according to any of the preceding claims containing up to 50 ppm by weight Se.
- 9. An IR and UV absorbing glass according to any of the preceding claims containing up to 200 ppm by weight ${\rm Co_30_4}$.

- 10. An IR and UV absorbing glass according to any of the preceding claims containing up to 2.5% by weight ${\rm Nd}_2{\rm O}_3$.
- 11. An IR and UV absorbing glass according to any of the preceding claims containing up to 200 ppm by weight NiO.
- 12. An IR and UV absorbing glass according to any of the preceding claims containing up to 1% by weight MnO.
- 13. An IR and UV absorbing glass having a neutral tint substantially as hereinbefore described in any one of the Examples.
- 14. An IR and UV absorbing glass according to any of the preceding claims in sheet form.
- 15. A window composed of glass according to any of the preceding claims.
- 16. An automotive window composed of glass according to any of the preceding claims.

· 	Figure 1 Gray glasses with improved DSHT	and
	UVT persorman	nce
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70	<u>a</u> 17	· · · · · · · · · · · · · · · · · · ·
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60	0547	
50		
2 40	1	
. X		
a 30	W	
20		
10		!
	0:05	0-15
GL		
Glass	LT DSHT UV A J* FeO S	Se 6,0, N1,0,
1	20 10 1/6 263 -1-1 0-35 0-63	11 41
3,	- 60 C1 23 - 1.4 - 6:35 6:682	3 32 075
	68 59 31 -0.8+2:0 6:38 6-8	5 39
	58 = 55 = 550 550 10 22/4 506 6238 76.599 2	2 32
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